Crystal structure of nonadecamethylated $6^A,6^C$-epithio-$6^A,6^C$-dideoxy-β-cyclodextrin — pentane — water (1:1:1), $C_{61}H_{106}O_{33}S \cdot C_5H_{12} \cdot H_2O$

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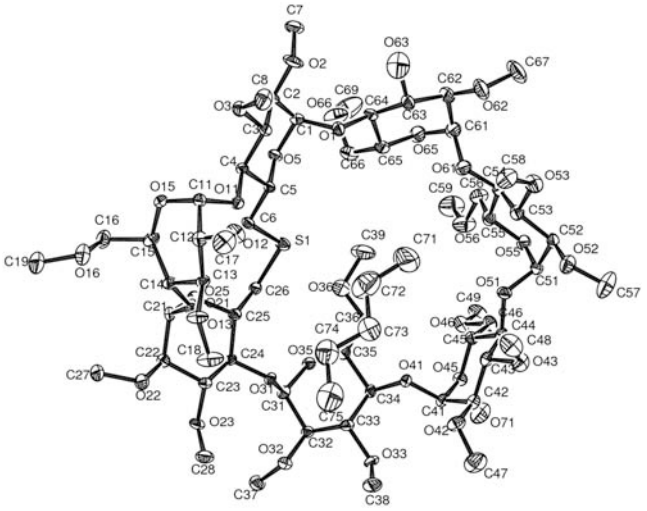

Abstract

$C_{66}H_{120}O_{34}S$, orthorhombic, $P2_12_12_1$ (no. 19),
a = 12.3247(5) Å, b = 15.4129(5) Å, c = 39.9464(9) Å,
V = 7588.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.073, $wR_{ref}(F^2)$ = 0.172,
T = 120 K.

Source of material

The title compound was prepared according to a method previously reported for related thia-capped cyclodextrins (CDs) by reaction of nonadecamethylated $6^A,6^C$-dimesyl-β-cyclodextrin with powdered, hydrated sodium sulfide ($Na_2S \cdot 9H_2O$) [1]. The product is water soluble.

Discussion

The cavity of the title crystal structure adopts an elongated shape, thereby considerably deviating from the usual torus shape of non-permethylated β-cyclodextrins (β-CDs) [2]. This can be seen, for example, when considering the distances between O21 and O51 (10.37 Å), and between O1 and O31 (8.53 Å). However, a similar lengthening was observed in the solid state of several permethylated β-CDs [3,4]. The presence of the sulfur cap pushes glucose ring B (i. e. the one lying between the capped sugar units) far away from the average plane defined by the O-4 atoms O1, O61, O51, O41, and O31 (distance of O15 to this plane 2.61 Å), all other glucose units being bisected by this plane. The sulfur bridge further induces a strong distortion within glucose ring B. Thus, while all other glucose rings are in the usual $^4C_1$ chair conformation, glucose B adopts a $^1H_2$ halfchair conformation. The sulfur atom lies above the cavity entrance, with one of its lone pairs pointing towards the interior of the receptor. Its distance to the plane defined above is 1.08 Å. A molecule of pentane is located inside the cavity, the C_5 chain being roughly parallel to the O31-O61 vector.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.28 × 0.32 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.30 cm^{-1}
Diffractometer, scan mode:	Saphire 3 Xcalibur CCD, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	55289, 16478
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 13768
$N(param)_{refined}$:	911
Programs:	SIR-97 [5], SHELXL-97 [6], PLATON [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(71D)	4*a*	0.0672	–0.3426	0.3474	0.050
H(71E)	4*a*	0.1585	–0.3567	0.3194	0.050
H(71A)	4*a*	–0.0645	0.1363	0.4263	0.166
H(71B)	4*a*	–0.1106	0.0421	0.4222	0.166
H(71C)	4*a*	–0.1820	0.1146	0.4387	0.166
H(72A)	4*a*	–0.1885	0.1879	0.3882	0.095
H(72B)	4*a*	–0.1162	0.1164	0.3717	0.095
H(73A)	4*a*	–0.3281	0.0880	0.3938	0.078
H(73B)	4*a*	–0.2557	0.0176	0.3765	0.078
H(74A)	4*a*	–0.3264	0.1698	0.3438	0.070
H(74B)	4*a*	–0.2581	0.0965	0.3266	0.070
H(75A)	4*a*	–0.4377	0.0826	0.3120	0.106
H(75B)	4*a*	–0.4705	0.0750	0.3498	0.106
H(75C)	4*a*	–0.4022	0.0012	0.3328	0.106
H(1)	4*a*	0.2087	0.4229	0.4092	0.026
H(2)	4*a*	0.1003	0.5434	0.3986	0.026
H(3)	4*a*	–0.0802	0.4478	0.4235	0.023
H(4)	4*a*	–0.0411	0.5037	0.3564	0.021
H(5)	4*a*	–0.0361	0.3304	0.3824	0.020
H(6A)	4*a*	0.0209	0.3818	0.3165	0.021
H(6B)	4*a*	–0.0903	0.3371	0.3250	0.021
H(7A)	4*a*	0.1983	0.5752	0.4771	0.060
H(7B)	4*a*	0.2134	0.6033	0.4396	0.060
H(7C)	4*a*	0.2427	0.5086	0.4508	0.060
H(8A)	4*a*	–0.1986	0.6324	0.4446	0.052
H(8B)	4*a*	–0.1233	0.5635	0.4618	0.052
H(8C)	4*a*	–0.2237	0.5332	0.4407	0.052
H(11)	4*a*	–0.2409	0.5585	0.3730	0.024
H(12)	4*a*	–0.4012	0.5344	0.3411	0.023
H(13)	4*a*	–0.3308	0.3598	0.3358	0.020
H(14)	4*a*	–0.3535	0.4505	0.2765	0.020
H(15)	4*a*	–0.1400	0.4723	0.2935	0.026
H(16A)	4*a*	–0.1657	0.6148	0.2724	0.034
H(16B)	4*a*	–0.1920	0.5402	0.2468	0.034

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(17A)	4*a*	–0.5340	0.4781	0.4097	0.058
H(17B)	4*a*	–0.5587	0.4748	0.3712	0.058
H(17C)	4*a*	–0.5081	0.5586	0.3870	0.058
H(18A)	4*a*	–0.5709	0.3066	0.2989	0.059
H(18B)	4*a*	–0.4679	0.2669	0.3161	0.059
H(18C)	4*a*	–0.4576	0.3130	0.2813	0.059
H(19A)	4*a*	–0.4049	0.6682	0.2322	0.067
H(19B)	4*a*	–0.3150	0.6117	0.2150	0.067
H(19C)	4*a*	–0.2829	0.6964	0.2344	0.067
H(21)	4*a*	–0.1865	0.4026	0.2439	0.020
H(22)	4*a*	–0.2094	0.2904	0.2060	0.021
H(23)	4*a*	–0.3086	0.1858	0.2561	0.020
H(24)	4*a*	–0.0966	0.1564	0.2299	0.019
H(25)	4*a*	–0.1468	0.2235	0.2950	0.021
H(26A)	4*a*	0.0653	0.2310	0.2706	0.024
H(26B)	4*a*	0.0279	0.1404	0.2840	0.024
H(27A)	4*a*	–0.4372	0.4074	0.1959	0.048
H(27B)	4*a*	–0.3282	0.3800	0.1787	0.048
H(27C)	4*a*	–0.3260	0.4376	0.2110	0.048
H(28A)	4*a*	–0.4166	0.0744	0.1937	0.047
H(28B)	4*a*	–0.4480	0.1577	0.2140	0.047
H(28C)	4*a*	–0.4065	0.0748	0.2328	0.047
H(31)	4*a*	–0.0759	0.0204	0.2278	0.021
H(32)	4*a*	–0.1253	–0.1224	0.2396	0.021
H(33)	4*a*	–0.2453	–0.0628	0.2955	0.022
H(34)	4*a*	–0.0500	–0.1605	0.2979	0.020
H(35)	4*a*	–0.0745	0.0187	0.3144	0.020
H(36A)	4*a*	0.0564	–0.0396	0.3483	0.030
H(36B)	4*a*	0.1124	–0.0857	0.3179	0.030
H(37A)	4*a*	–0.3181	–0.0668	0.1851	0.040
H(37B)	4*a*	–0.2209	–0.1305	0.1912	0.040
H(37C)	4*a*	–0.2000	–0.0303	0.1888	0.040
H(38A)	4*a*	–0.3920	–0.2425	0.2886	0.052
H(38B)	4*a*	–0.3952	–0.1503	0.2721	0.052
H(38C)	4*a*	–0.3782	–0.1593	0.3108	0.052
H(39A)	4*a*	0.2538	0.1020	0.3449	0.081
H(39B)	4*a*	0.2537	0.0022	0.3525	0.081
H(39C)	4*a*	0.1643	0.0620	0.3681	0.081
H(41)	4*a*	–0.1488	–0.2531	0.3314	0.022
H(42)	4*a*	–0.1978	–0.2925	0.3856	0.025
H(43)	4*a*	–0.1589	–0.1195	0.4063	0.024
H(44)	4*a*	–0.0319	–0.2634	0.4278	0.023
H(45)	4*a*	0.0447	–0.1399	0.3802	0.022

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(46A)	4*a*	0.1786	–0.2181	0.4111	0.029
H(46B)	4*a*	0.1333	–0.3050	0.3960	0.029
H(47A)	4*a*	–0.4484	–0.2456	0.3661	0.071
H(47B)	4*a*	–0.3807	–0.2989	0.3922	0.071
H(47C)	4*a*	–0.3575	–0.3092	0.3538	0.071
H(48A)	4*a*	–0.3192	–0.1539	0.4713	0.064
H(48B)	4*a*	–0.3248	–0.1102	0.4359	0.064
H(48C)	4*a*	–0.2272	–0.0902	0.4599	0.064
H(49A)	4*a*	0.3520	–0.2657	0.3467	0.051
H(49B)	4*a*	0.2975	–0.3354	0.3698	0.051
H(49C)	4*a*	0.3475	–0.2512	0.3855	0.051
H(51)	4*a*	0.0631	–0.2284	0.4717	0.026
H(52)	4*a*	0.0778	–0.1363	0.5191	0.027
H(53)	4*a*	–0.0133	–0.0032	0.4778	0.027
H(54)	4*a*	0.2032	–0.0037	0.5028	0.030
H(55)	4*a*	0.1474	–0.0363	0.4346	0.029
H(56A)	4*a*	0.3273	0.0215	0.4446	0.039
H(56B)	4*a*	0.3539	–0.0649	0.4639	0.039
H(57A)	4*a*	–0.1683	–0.2322	0.5268	0.078
H(57B)	4*a*	–0.0506	–0.2304	0.5417	0.078
H(57C)	4*a*	–0.0702	–0.2652	0.5053	0.078
H(58A)	4*a*	–0.0723	0.1176	0.5434	0.055
H(58B)	4*a*	–0.1250	0.0725	0.5122	0.055
H(58C)	4*a*	–0.0252	0.1343	0.5075	0.055
H(59A)	4*a*	0.4495	–0.0969	0.3838	0.090
H(59B)	4*a*	0.4875	–0.0899	0.4211	0.090
H(59C)	4*a*	0.4497	–0.0066	0.4018	0.090
H(61)	4*a*	0.2950	0.1105	0.4933	0.037
H(62)	4*a*	0.2847	0.2557	0.5072	0.036
H(63)	4*a*	0.0695	0.2662	0.4823	0.029
H(64)	4*a*	0.2478	0.3430	0.4487	0.026
H(65)	4*a*	0.1501	0.1869	0.4252	0.030
H(66A)	4*a*	0.3033	0.1819	0.3886	0.037
H(66B)	4*a*	0.2240	0.2585	0.3803	0.037
H(67A)	4*a*	0.1899	0.1576	0.5766	0.086
H(67B)	4*a*	0.2903	0.2073	0.5619	0.086
H(67C)	4*a*	0.2596	0.1153	0.5481	0.086
H(68A)	4*a*	0.0489	0.4524	0.5241	0.074
H(68B)	4*a*	0.0117	0.3556	0.5277	0.074
H(68C)	4*a*	–0.0076	0.4053	0.4940	0.074
H(69A)	4*a*	0.5166	0.3080	0.4013	0.113
H(69B)	4*a*	0.4744	0.2363	0.3767	0.113
H(69C)	4*a*	0.4740	0.2194	0.4154	0.113

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(71)	4*a*	0.0940(3)	–0.3954(2)	0.3305(1)	0.059(2)	0.039(2)	0.053(2)	–0.007(2)	0.014(2)	–0.001(2)
C(71)	4*a*	–0.1285(9)	0.1027(5)	0.4219(2)	0.148(9)	0.070(5)	0.115(7)	–0.008(6)	–0.083(7)	0.018(5)
C(72)	4*a*	–0.1726(6)	0.1263(5)	0.3882(2)	0.067(5)	0.053(4)	0.117(7)	0.000(3)	–0.001(5)	0.002(4)
C(73)	4*a*	–0.2715(6)	0.0792(5)	0.3773(2)	0.079(5)	0.063(4)	0.053(4)	–0.011(4)	–0.009(3)	0.006(3)
C(74)	4*a*	–0.3133(6)	0.1078(4)	0.3434(2)	0.063(4)	0.057(4)	0.054(4)	0.005(3)	0.007(3)	0.001(3)
C(75)	4*a*	–0.4149(6)	0.0626(5)	0.3336(2)	0.082(5)	0.062(4)	0.068(4)	0.008(4)	0.017(4)	–0.004(3)
S(1)	4*a*	0.04947(9)	0.23538(6)	0.32831(3)	0.0285(5)	0.0192(4)	0.0274(5)	0.0063(4)	–0.0131(4)	–0.0043(4)
O(1)	4*a*	0.0984(2)	0.3503(2)	0.43057(7)	0.021(1)	0.023(1)	0.020(1)	–0.002(1)	–0.005(1)	0.006(1)
O(2)	4*a*	0.0867(3)	0.5296(2)	0.44709(8)	0.045(2)	0.035(2)	0.026(2)	0.003(1)	–0.018(2)	–0.014(1)
O(3)	4*a*	–0.1013(2)	0.5728(2)	0.41339(6)	0.028(2)	0.019(1)	0.015(1)	0.003(1)	0.004(1)	–0.002(1)
O(5)	4*a*	0.1066(2)	0.3869(2)	0.37381(6)	0.018(1)	0.024(1)	0.016(1)	–0.003(1)	–0.004(1)	–0.002(1)
O(11)	4*a*	–0.1810(2)	0.4421(2)	0.36541(7)	0.015(1)	0.016(1)	0.023(1)	–0.002(1)	–0.006(1)	0.002(1)
O(12)	4*a*	–0.4041(2)	0.4632(2)	0.38191(7)	0.022(2)	0.040(2)	0.023(2)	0.002(1)	0.001(1)	0.000(1)
O(13)	4*a*	–0.4744(2)	0.3947(2)	0.31900(7)	0.015(1)	0.026(1)	0.033(2)	–0.003(1)	–0.000(1)	–0.013(1)
O(15)	4*a*	–0.2068(2)	0.5530(2)	0.32544(7)	0.032(2)	0.014(1)	0.020(1)	–0.006(1)	–0.004(1)	0.001(1)
O(16)	4*a*	–0.3193(3)	0.5981(2)	0.26443(8)	0.042(2)	0.028(2)	0.035(2)	0.004(1)	–0.000(2)	0.010(1)
O(21)	4*a*	–0.2582(2)	0.3485(2)	0.28256(6)	0.029(2)	0.013(1)	0.018(1)	0.004(1)	0.003(1)	0.001(1)
O(22)	4*a*	–0.3594(2)	0.3151(2)	0.21988(7)	0.022(1)	0.021(1)	0.026(2)	0.004(1)	–0.002(1)	0.002(1)
O(23)	4*a*	–0.2918(2)	0.1395(2)	0.21018(7)	0.027(2)	0.023(1)	0.019(1)	–0.002(1)	–0.008(1)	–0.006(1)
O(25)	4*a*	–0.0936(2)	0.3082(2)	0.26054(6)	0.020(1)	0.013(1)	0.019(1)	0.001(1)	–0.002(1)	–0.001(1)
O(31)	4*a*	–0.1611(2)	0.0758(2)	0.26347(6)	0.020(1)	0.009(1)	0.017(1)	0.001(1)	0.002(1)	–0.0016(9)
O(32)	4*a*	–0.2654(2)	–0.0664(2)	0.23165(7)	0.020(1)	0.023(1)	0.019(1)	–0.001(1)	–0.002(1)	–0.002(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(33)	4*a*	–0.2483(2)	–0.1881(2)	0.28453(7)	0.023(1)	0.016(1)	0.030(2)	–0.008(1)	–0.003(1)	–0.001(1)
O(35)	4*a*	–0.0046(2)	–0.0088(2)	0.27071(7)	0.017(1)	0.016(1)	0.023(1)	0.001(1)	–0.002(1)	–0.001(1)
O(36)	4*a*	0.1413(3)	0.0377(2)	0.32039(9)	0.041(2)	0.042(2)	0.043(2)	–0.007(2)	–0.013(2)	0.005(2)
O(41)	4*a*	–0.1223(2)	–0.1293(2)	0.34062(6)	0.022(1)	0.013(1)	0.017(1)	–0.001(1)	–0.001(1)	0.002(1)
O(42)	4*a*	–0.3024(2)	–0.2020(2)	0.37276(8)	0.017(1)	0.023(1)	0.039(2)	–0.001(1)	0.002(1)	–0.002(1)
O(43)	4*a*	–0.2190(2)	–0.2045(2)	0.43875(7)	0.029(2)	0.035(2)	0.026(2)	0.000(1)	0.011(1)	–0.001(1)
O(45)	4*a*	–0.0150(2)	–0.2465(2)	0.35878(7)	0.020(1)	0.020(1)	0.022(1)	0.004(1)	–0.002(1)	–0.002(1)
O(46)	4*a*	0.2083(2)	–0.2317(2)	0.36308(7)	0.025(2)	0.034(2)	0.023(1)	0.006(1)	0.001(1)	0.001(1)
O(51)	4*a*	–0.0013(2)	–0.1414(2)	0.44200(7)	0.026(2)	0.018(1)	0.021(1)	0.001(1)	–0.005(1)	–0.004(1)
O(52)	4*a*	–0.0781(3)	–0.1393(2)	0.50893(8)	0.034(2)	0.028(2)	0.031(2)	–0.003(1)	0.008(1)	0.004(1)
O(53)	4*a*	0.0134(3)	0.0202(2)	0.52586(8)	0.037(2)	0.031(2)	0.027(2)	0.008(1)	0.003(1)	–0.006(1)
O(55)	4*a*	0.1768(2)	–0.1420(2)	0.46216(7)	0.024(2)	0.019(1)	0.028(2)	0.004(1)	–0.003(1)	0.002(1)
O(56)	4*a*	0.3316(3)	–0.0819(2)	0.41539(9)	0.037(2)	0.053(2)	0.042(2)	–0.010(2)	0.011(2)	–0.017(2)
O(61)	4*a*	0.1478(2)	0.0948(2)	0.47586(8)	0.027(2)	0.020(1)	0.040(2)	0.001(1)	–0.014(1)	0.003(1)
O(62)	4*a*	0.1646(3)	0.2074(2)	0.53260(8)	0.042(2)	0.057(2)	0.027(2)	–0.007(2)	–0.011(2)	0.015(2)
O(63)	4*a*	0.1429(3)	0.3700(2)	0.50085(8)	0.037(2)	0.033(2)	0.027(2)	0.011(1)	–0.005(1)	–0.008(1)
O(65)	4*a*	0.2880(2)	0.1717(2)	0.45034(8)	0.026(2)	0.026(2)	0.041(2)	0.003(1)	–0.009(1)	0.004(1)
O(66)	4*a*	0.3613(3)	0.2964(2)	0.4004(1)	0.032(2)	0.035(2)	0.061(2)	–0.002(2)	0.002(2)	–0.018(2)
C(1)	4*a*	0.1304(3)	0.4136(2)	0.4072(1)	0.023(2)	0.021(2)	0.021(2)	–0.007(2)	–0.005(2)	–0.000(2)
C(2)	4*a*	0.0717(3)	0.4995(2)	0.4140(1)	0.029(2)	0.021(2)	0.016(2)	–0.007(2)	–0.009(2)	–0.002(2)
C(3)	4*a*	–0.0501(3)	0.4906(2)	0.40789(9)	0.026(2)	0.018(2)	0.013(2)	–0.001(2)	–0.002(2)	–0.001(1)
C(4)	4*a*	–0.0682(3)	0.4600(2)	0.37216(9)	0.019(2)	0.020(2)	0.014(2)	–0.007(1)	–0.002(1)	0.001(1)
C(5)	4*a*	–0.0070(3)	0.3743(2)	0.36709(9)	0.016(2)	0.020(2)	0.014(2)	–0.006(1)	–0.004(1)	0.003(1)
C(6)	4*a*	–0.0147(3)	0.3414(2)	0.33154(9)	0.018(2)	0.018(2)	0.017(2)	0.003(1)	–0.006(1)	–0.002(1)
C(7)	4*a*	0.1938(4)	0.5563(3)	0.4542(1)	0.052(3)	0.028(2)	0.040(3)	–0.002(2)	–0.028(2)	–0.006(2)
C(8)	4*a*	–0.1670(4)	0.5757(3)	0.4425(1)	0.048(3)	0.029(2)	0.027(2)	0.002(2)	0.013(2)	0.000(2)
C(11)	4*a*	–0.2429(3)	0.5148(2)	0.3552(1)	0.025(2)	0.015(2)	0.020(2)	0.001(2)	–0.006(2)	0.000(1)
C(12)	4*a*	–0.3595(3)	0.4858(2)	0.3503(1)	0.017(2)	0.019(2)	0.022(2)	0.004(2)	0.001(2)	–0.002(2)
C(13)	4*a*	–0.3628(3)	0.4113(2)	0.32538(9)	0.016(2)	0.013(2)	0.022(2)	0.001(1)	–0.002(2)	–0.001(1)
C(14)	4*a*	–0.3006(3)	0.4315(2)	0.29329(9)	0.018(2)	0.013(2)	0.020(2)	0.001(1)	–0.000(2)	–0.004(1)
C(15)	4*a*	–0.2098(3)	0.5023(2)	0.2953(1)	0.018(2)	0.016(2)	0.031(2)	0.000(2)	0.004(2)	–0.002(2)
C(16)	4*a*	–0.2147(4)	0.5671(3)	0.2676(1)	0.036(2)	0.024(2)	0.026(2)	–0.002(2)	0.004(2)	0.007(2)
C(17)	4*a*	–0.5098(4)	0.4963(3)	0.3879(1)	0.035(3)	0.042(3)	0.038(3)	0.001(2)	0.016(2)	–0.003(2)
C(18)	4*a*	–0.4944(4)	0.3136(3)	0.3025(1)	0.022(2)	0.033(2)	0.062(3)	0.000(2)	–0.005(2)	–0.022(2)
C(19)	4*a*	–0.3316(5)	0.6477(3)	0.2340(1)	0.053(3)	0.030(2)	0.051(3)	0.001(2)	–0.012(3)	0.015(2)
C(21)	4*a*	–0.1961(3)	0.3440(2)	0.25295(9)	0.019(2)	0.017(2)	0.014(2)	0.001(1)	–0.000(1)	0.004(1)
C(22)	4*a*	–0.2515(3)	0.2870(2)	0.22681(9)	0.017(2)	0.017(2)	0.018(2)	0.003(1)	–0.001(2)	–0.002(1)
C(23)	4*a*	–0.2568(3)	0.1917(2)	0.23768(9)	0.017(2)	0.017(2)	0.016(2)	–0.002(1)	–0.001(2)	–0.003(1)
C(24)	4*a*	–0.1458(3)	0.1602(2)	0.24914(9)	0.020(2)	0.010(2)	0.017(2)	–0.000(1)	0.000(2)	–0.002(1)
C(25)	4*a*	–0.0994(3)	0.2223(2)	0.27529(9)	0.021(2)	0.014(2)	0.019(2)	0.002(1)	–0.004(2)	–0.004(1)
C(26)	4*a*	0.0160(3)	0.2024(2)	0.2860(1)	0.023(2)	0.012(2)	0.025(2)	0.002(1)	–0.005(2)	–0.000(2)
C(27)	4*a*	–0.3630(4)	0.3912(3)	0.1997(1)	0.033(2)	0.025(2)	0.037(2)	–0.002(2)	–0.014(2)	0.011(2)
C(28)	4*a*	–0.3993(4)	0.1092(3)	0.2129(1)	0.027(2)	0.026(2)	0.040(3)	–0.002(2)	–0.015(2)	–0.004(2)
C(31)	4*a*	–0.0986(3)	0.0064(2)	0.2507(1)	0.020(2)	0.013(2)	0.020(2)	0.003(1)	0.003(2)	–0.003(1)
C(32)	4*a*	–0.1680(3)	–0.0763(2)	0.25012(9)	0.017(2)	0.014(2)	0.022(2)	0.001(1)	–0.004(2)	–0.001(1)
C(33)	4*a*	–0.1962(3)	–0.1053(2)	0.28533(9)	0.020(2)	0.017(2)	0.019(2)	–0.003(2)	–0.001(2)	–0.004(1)
C(34)	4*a*	–0.0950(3)	–0.1129(2)	0.30638(9)	0.020(2)	0.012(2)	0.017(2)	0.004(1)	–0.001(2)	0.001(1)
C(35)	4*a*	–0.0300(3)	–0.0282(2)	0.3053(1)	0.017(2)	0.012(2)	0.022(2)	0.002(1)	0.001(2)	0.001(1)
C(36)	4*a*	0.0736(3)	–0.0338(3)	0.3247(1)	0.020(2)	0.022(2)	0.032(2)	–0.003(2)	–0.009(2)	0.004(2)
C(37)	4*a*	–0.2498(4)	–0.0741(3)	0.1963(1)	0.029(2)	0.031(2)	0.022(2)	–0.003(2)	–0.001(2)	–0.001(2)
C(38)	4*a*	–0.3628(4)	–0.1848(3)	0.2894(1)	0.023(2)	0.042(3)	0.038(3)	–0.015(2)	–0.004(2)	0.002(2)
C(39)	4*a*	0.2087(5)	0.0521(4)	0.3488(2)	0.051(3)	0.049(3)	0.061(4)	–0.002(3)	–0.027(3)	–0.017(3)
C(41)	4*a*	–0.1217(3)	–0.2179(2)	0.3501(1)	0.017(2)	0.014(2)	0.024(2)	0.000(1)	0.001(2)	0.003(1)
C(42)	4*a*	–0.1953(3)	–0.2305(2)	0.3802(1)	0.020(2)	0.018(2)	0.024(2)	0.001(2)	0.004(2)	0.002(2)
C(43)	4*a*	–0.1534(3)	–0.1821(2)	0.4104(1)	0.020(2)	0.018(2)	0.023(2)	0.001(2)	0.002(2)	0.002(2)
C(44)	4*a*	–0.0360(3)	–0.2052(2)	0.4180(1)	0.020(2)	0.016(2)	0.022(2)	0.002(1)	0.000(2)	0.002(1)
C(45)	4*a*	0.0338(3)	–0.2008(2)	0.38644(9)	0.021(2)	0.018(2)	0.016(2)	0.002(2)	–0.001(2)	0.000(1)
C(46)	4*a*	0.1431(3)	–0.2435(3)	0.3918(1)	0.021(2)	0.030(2)	0.022(2)	0.005(2)	0.000(2)	0.002(2)
C(47)	4*a*	–0.3782(4)	–0.2692(3)	0.3711(2)	0.024(2)	0.045(3)	0.073(4)	–0.004(2)	–0.007(3)	0.008(3)
C(48)	4*a*	–0.2772(4)	–0.1340(4)	0.4526(1)	0.035(3)	0.058(3)	0.035(3)	0.010(2)	0.009(2)	–0.007(2)
C(49)	4*a*	0.3095(4)	–0.2745(3)	0.3666(1)	0.022(2)	0.051(3)	0.030(2)	0.009(2)	–0.002(2)	–0.007(2)
C(51)	4*a*	0.0675(3)	–0.1655(2)	0.4684(1)	0.028(2)	0.015(2)	0.023(2)	0.001(2)	–0.007(2)	0.002(2)
C(52)	4*a*	0.0302(4)	–0.1195(2)	0.5005(1)	0.031(2)	0.020(2)	0.018(2)	0.001(2)	–0.003(2)	0.003(2)
C(53)	4*a*	0.0390(4)	–0.0211(2)	0.4950(1)	0.028(2)	0.018(2)	0.021(2)	0.005(2)	0.000(2)	–0.001(2)
C(54)	4*a*	0.1531(4)	0.0045(3)	0.4840(1)	0.027(2)	0.022(2)	0.026(2)	0.001(2)	–0.008(2)	0.003(2)
C(55)	4*a*	0.1907(3)	–0.0509(2)	0.4544(1)	0.023(2)	0.023(2)	0.027(2)	0.004(2)	–0.004(2)	0.006(2)
C(56)	4*a*	0.3098(4)	–0.0397(3)	0.4463(1)	0.032(3)	0.030(2)	0.035(2)	–0.006(2)	0.002(2)	–0.006(2)
C(57)	4*a*	–0.0930(5)	–0.2234(3)	0.5217(2)	0.060(4)	0.032(3)	0.064(4)	–0.002(2)	0.026(3)	0.013(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(58)	4*a*	–0.0581(4)	0.0920(3)	0.5219(1)	0.031(3)	0.031(2)	0.047(3)	0.003(2)	0.007(2)	–0.004(2)
C(59)	4*a*	0.4378(5)	–0.0677(5)	0.4047(2)	0.044(3)	0.076(4)	0.059(4)	–0.015(3)	0.019(3)	–0.015(3)
C(61)	4*a*	0.2417(4)	0.1458(3)	0.4812(1)	0.027(2)	0.025(2)	0.042(3)	–0.000(2)	–0.016(2)	0.008(2)
C(62)	4*a*	0.2162(4)	0.2262(3)	0.5023(1)	0.028(2)	0.035(2)	0.028(2)	–0.004(2)	–0.013(2)	0.006(2)
C(63)	4*a*	0.1435(3)	0.2895(3)	0.4831(1)	0.023(2)	0.028(2)	0.021(2)	–0.002(2)	–0.007(2)	0.005(2)
C(64)	4*a*	0.1841(3)	0.3051(2)	0.4477(1)	0.021(2)	0.022(2)	0.021(2)	–0.003(2)	–0.008(2)	–0.001(2)
C(65)	4*a*	0.2156(3)	0.2211(3)	0.4296(1)	0.023(2)	0.025(2)	0.027(2)	–0.004(2)	–0.009(2)	0.000(2)
C(66)	4*a*	0.2749(4)	0.2366(3)	0.3968(1)	0.033(2)	0.034(2)	0.025(2)	–0.002(2)	–0.004(2)	–0.009(2)
C(67)	4*a*	0.2313(6)	0.1689(4)	0.5567(1)	0.076(4)	0.062(4)	0.033(3)	0.012(3)	–0.026(3)	0.005(3)
C(68)	4*a*	0.0408(5)	0.3980(4)	0.5126(2)	0.051(3)	0.046(3)	0.051(3)	0.019(3)	0.017(3)	–0.002(3)
C(69)	4*a*	0.4644(6)	0.2625(4)	0.3983(2)	0.055(4)	0.054(4)	0.117(6)	–0.016(3)	0.040(4)	–0.021(4)

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